

ACOUSTIC EMISSION TESTING
OF COMPOSITE VESSELS UNDER
SUSTAINED LOADING

R. F. Lark and P. E. Moorhead
Lewis Research Center
Cleveland, Ohio

DISTRIBUTION STATEMENT A

Approved for public release;
Distribution Unlimited

19960228 016

TECHNICAL PAPER to be presented at the
Symposium on Nondestructive Evaluation and Flaw Criticality
for Composite Materials
sponsored by the American Society for Testing and Materials
Philadelphia, Pennsylvania, October 10-11, 1978

DEPARTMENT OF DEFENSE
PLASTICS TECHNICAL EVALUATION CENTER
ARRADCOM, DOVER, N. J. 07301

DO NOT QUALIFY INFORMATION

PLASTEC 32589

ACOUSTIC EMISSION TESTING OF COMPOSITE VESSELS

UNDER SUSTAINED LOADING

by R. F. Lark and P. E. Moorhead

INTRODUCTION

Filament-wound (FW) composite pressure vessels constructed from advanced high-strength-to-density and modulus-to-density ratio fiber/resin materials provide significant weight savings over high performance, all-metal pressure vessels for containment of high pressure gases and fluids. The structural efficiencies of the all-metal pressure vessels made from aluminum or titanium alloys range from 0.76 to 1.52×10^6 cm based on a pressure vessel performance factor ($P_b V/W$) where P_b is the burst pressure, V is the contained volume, and W is the vessel weight. FW composite pressure vessels are capable of yielding $P_b V/W$ values ranging from 2.03 to 3.05×10^6 cm when made from the advanced fiber/resin composites and equipped with metallic load-bearing or thin metal liners. Consequently, FW vessels have the capability of providing a weight savings of 25-percent or more over conventional metallic pressure vessels. Other attractive features of the composite pressure vessels are a medium-to-high cyclic life capability, zero permeability, chemical compatibility with high pressure gases and fluids and a leak-before-fracture failure mode (Ref. 1).

During the development of FW composite pressure vessel technology, prototype vessels of various designs and materials are routinely subjected to single-cycle burst, cyclic, and sustained load tests to determine vessel structural efficiencies and other operational characteristics. No non-destructive test method is, however, currently available to determine

the integrity or predict the burst pressure of composite pressure vessels. A suitable nondestructive test method would aid in improving the quality control of the vessel winding process, certifying the strength of a virgin vessel, and possibly predicting residual service life.

One nondestructive test method involves the study of the low-level acoustic or stress wave emissions generated by materials under stress. Exploratory programs have been sponsored by NASA, DOE (and other agencies) for studying the acoustic emissions (AE) generated by advanced fiber/resin composite pressure vessels under various loading environments. These programs have shown that the AE test method has potential for prediction of virgin and in-service vessel integrity. The objective of this paper is to present the findings of a NASA LeRC program that studied the AE signals from small diameter Kevlar 49/epoxy pressure vessels subjected to long-term sustained loading tests. In addition single cycle burst tests were conducted to provide a basis for calculating the test pressure used in the sustained loading tests.

MATERIALS AND EXPERIMENTAL PROCEDURE

Composite pressure vessel design and fabrication. - The composite pressure vessels used in this program were fabricated by the Lawrence Livermore Laboratory under NASA/DOE Interagency Agreement C-13980-C. The pressure vessels were fabricated using a numerically controlled filament-winding machine. Prior to the winding of the vessels, Kevlar 49 fibers were impregnated with controlled quantities of an epoxy matrix resin by passing the fiber into a vacuum chamber controlled at approximately 5 mm Hg. The resin was metered and applied directly to the fibers in the vacuum chamber. The resin impregnated fibers then exited

the vacuum chamber and were led to the winding machine where they were wound directly over an elastomer coated wash-out type mandrel. After completion of winding, the vessels were cured. The mandrels were removed by a high pressure water wash. The design and dimensions of the vessels are shown in Fig. 1. The fiber and resin materials used for fabrication of the vessels along with the curing procedure is shown on Table 1. The vessels were designed to have a single-cycle burst strength of 2500 psig. The fiber volumes in the Kevlar 49/epoxy resin composite ranged from 67.7 to 73.0-percent and averaged 70.3-percent.

Hydraulic pressurization apparatus. - Each vessel was mounted in a test fixture that isolated the specimen from external noises and/or vibrations during the single-cycle burst and sustained loading tests. A view of the test fixture is shown in Fig. 2. After the vessel and AE transducers were installed, an enclosure (not shown) was positioned around the vessel to provide further isolation and to collect hydraulic fluid upon vessel failure. The vessels were pressurized using an ethylene glycol/water solution. The hydraulic pressurization apparatus, shown in Fig. 3, provided a uniform, repeatable pressure ramp (0.0048 GPa/min) during all tests. A servo-controlled valve on the pressurization apparatus was controlled by an external ramp generator. For the sustained loading tests, the vessels were isolated from the pressurization apparatus after pressure was stabilized during the initial pressurization cycle. Changes in vessel pressure during these tests were minimized by using a hydraulic accumulator (not shown) in the vessel piping system. In addition, the ambient environment in the test cell

was controlled to provide a constant temperature. Occasional minor leakage of hydraulic fluid from the pressurization system would tend to reduce system pressure which was then corrected, as needed, by using a hand pump.

Vessel pressure during the single-cycle burst tests was recorded on a strip chart recorder. Vessel pressure during the sustained-load tests was monitored visually on a calibrated pressure gage. The test duration was recorded by a pressure-actuated electrical timer.

Acoustic emission instrumentation. - A commercially available multi-channel AE apparatus was utilized and is shown in Fig. 4. The AE transducers used were of the differential piezo-electric type (PZT-5) with a resonant frequency of approximately 150 KHz. Figure 2 shows, in general fashion, how the transducers were attached to the vessels. The top and bottom transducers were mounted to the vessel bosses by means of the adapter plate shown in Fig. 5. Figure 5(a) shows an end view of a vessel and boss. An aluminum adapter plate was fastened to the metallic vessel boss by means of an interference fit. Figure 5(b) shows in cross sectional view how the adapter plate was attached to the bosses. The middle transducer was mounted to the composite vessel wall by means of a cylindrical shoe that was shaped to the contour of the vessel at one end and flat on the other end to match the transducer surface. A viscous polymeric couplant material was used to provide acoustic coupling between the three transducers and the boss adapter and shoe parts. All three transducers were held in place on their respective adapters or shoes by elastomeric bands. A fourth transducer of the same type was positioned in close proximity to the vessel without con-

tacting either the vessel surface or vessel support surface to monitor external noises, vibrations or electrical signals that might occur during the tests.

All of the AE transducers were initially selected for uniformity on the basis of uniform frequency response plots (furnished by the manufacturer). The AE count sensitivities of all of the transducers were then matched prior to the vessel tests by mounting the transducers, in pairs, on a plate that simulated the composite vessel. A simulated AE event, provided by an electrical discharge device, was introduced mid-way between the transducers. The gains of the AE count totalizers were adjusted to provide identical AE counts for a given event. This procedure was repeated until all of the AE transducers were calibrated against each other. After the transducers were mounted on the vessels, the same electrical discharge device was used to simulate an AE event mid-way on the hoop section of the vessels. It was found that only minute gain adjustments were required in the AE count totalizers to provide excellent matching of the top and bottom boss-mounted transducers. This procedure did not provide further checks on the matching of the wall-mounted and control transducers. After each vessel test, the AE count sensitivities of the boss mounted transducers were checked in place on the next vessel to be tested and were found to be identical with the first calibrations. The wall mounted and the control transducers were checked by using the plate mounting procedure described above. The AE count sensitivities of the later transducers were also found to be identical with the first calibrations.

The AE signals from each of the transducers were amplified (40 dB) by the preamplifiers in each channel. The preamplified signals were further amplified (37 to 39 dB) and subsequently processed for AE rate and count summation in the signal processing sections of the system. The total amplification of the AE signals was adjusted to give maximum amplification without extraneous interference from background electrical and other environmental noises. The AE system filters were adjusted to pass all frequencies greater than 0.1 MHz. The AE rate and total count data for each channel were recorded on the 'XYY' plotters on a continuous basis.

RESULTS AND DISCUSSION

Vessel burst strength tests. - The single cycle vessel burst strength tests were conducted to provide a basis for calculating the test pressure to be used for the sustained load tests. The data from single-cycle burst tests of 4-inch diameter, Kevlar 49/epoxy composite vessels are shown in Table 2. The average burst strength, based on four vessels, was 0.0163 GPa (2400 psi). The variation in this data was only 3.3 percent. Although these vessels were equipped with AE transducers, the data were not presented because of the wide scatter caused by a nonuniform pressurization ramp used during the burst test. These tests were conducted prior to the installation of the controlled ramp pressurization apparatus used for the remaining vessels in the program.

Sustained load/acoustic emission tests. - Kevlar 49/epoxy vessels, identical in design to those used for the single-cycle burst strength tests, were used to conduct the sustained load tests. These vessels were subjected to sustained load at 90-percent of the average single-cycle burst

strengths, or 2160 psi. During these tests the vessels were pressurized by a controlled ramp pressurization apparatus from zero pressure to 0.0147 GPa (2160 psi). This pressure was then maintained on a continuous basis until vessel failure occurred. A view of a typical vessel after failure is shown in Fig. 6. Failures occurred in the hoop section of all of the vessels. The test results in Figs. 7 to 13 show the summation of AE counts vs sustained load time to failure. In each of these figures the data represent AE signals sensed by the bottom transducer. The data for the AE signals sensed by the top boss transducers are similar. The AE test data and the times for vessel failure are shown in Table 3.

Virtually no AE counts were recorded on the control AE transducers during the tests. The AE data therefore did not require correction for environmental noise. As shown in Table 3 the AE counts sensed by the transducers located on the vessel bosses were averaged and yielded values that were relatively similar to each other and ranged from 36 to 53 K counts. The AE counts from the transducers mounted on the side wall of the composite vessels, on the other hand, varied significantly from 169 to 501 K counts. These large variations are believed to be due to signal attenuations resulting from AE events that may be near or distant from the transducer location. In the case of two AE events that yield similar energies, the event occurring close to the transducer would be sensed as an event having a greater energy than is the case of an AE event that is distant from the transducer. The close proximity AE event would result in a larger transducer voltage output and would, therefore, be recorded as having a greater count number than the distant AE event. Hamstad (Ref. 2) conducted AE tests of Kevlar 49/epoxy vessels equipped

with wall-mounted transducers. He concluded that an AE system should be operated using a relatively low frequency bandpass filter in order to avoid the large signal propagation losses encountered by signals processed at high frequency levels (<150 KHz) in a conventional AE system. The use of low frequency bandpass filters may, however, present a noise control problem since some of the signal frequencies being processed are in the human audible range. The use of a bandpass filter that passes only 20 KHz and higher frequencies might be a reasonable compromise between signal transmission and interference.

The close similarity of the average AE signals sensed by the top and bottom transducers indicates that the AE signals from these transducers represent an "integrated average" of the emissions generated by all of the failure processes, such as ply movement, matrix resin cracking, and fiber fractures, that occur during the sustained load to-failure tests. Based on work conducted at the Lawrence Livermore Laboratory by Hamstad (Refs. 3, 4, and 5) on fiber/epoxy composite pressure vessel tests, the source of 90-percent of the AE signals generated was associated with fiber fractures. The majority of the AE signal activity observed for the vessels in this program subjected to sustained load-to-failure tests are, accordingly, believed to be due to fiber fractures with a minor contribution from resin cracking and ply movement.

The close similarity of the AE data from the boss-mounted transducers also indicates that a certain critical number of Kevlar 49 fibers must fracture prior to initiation of vessel failure. This was also observed in an exploratory program by Hoggatt (Ref. 6) in which PRD-49-1 (Kevlar 49) fiber/epoxy composite vessels were subjected to single-cycle

burst strength and failure tests. These vessels were equipped with boss-mounted AE transducers. The total AE counts for vessels subjected to single-cycle burst as well as to cyclic fatigue were found to be comparable.

It was found that the AE data from the vessels tested in this program were independent of time insofar as vessel failure was concerned. This characteristic agrees with the concept that a minimum numbers of fibers must fracture prior to vessel failure.

Figure 7 also shows the typical summation of AE counts generated during the total test period. During the initial 5 minute pressurization period the AE rate activity was high and the total counts are plotted directly on the ordinate. This initial, period is referred to as the "shake-down" period. This initial high rate of AE activity was observed for all of the vessels. Next is shown a period of time (48 h) in which the AE count rate increased until stabilization occurred. Next follows a stabilized, active, stabilized, active, and stabilized period (740 h). This relatively low-AE rate period is called the "induction" period. This is followed by a high-rate AE count period that occurred prior to vessel failure. In five of the vessels tested, the low-rate AE "induction" periods and the high rate AE periods prior to vessel failure are observed (Figs. 7, 8, 10, 11, and 12). Two of the seven vessels showed a relatively uniform AE count rate commencing from the initial 5 minute shake-down pressurization period until vessel failure occurred (Figs. 9 and 13). The reason for these two anomalous data points may be due to low material strength or fabrication deficiencies.

It is postulated that the absence of significant AE counts during the low-rate count or "induction" period shown for these vessels (Figs. 7, 8, 10, 11, and 12) indicates the absence of significant fiber fractures. This has been observed by Chiao et al. in Refs. 7 and 8 in strength retention studies of Kevlar 49 and S-glass/epoxy strand specimens subjected to sustained loading tests. Strand specimens, subjected to sustained loading under varying percentages of ultimate strength, were periodically withdrawn and tested for ultimate strength. These studies showed no significant loss of tensile strength before the specimens failed in the sustained load tests.

Figure 14 shows a summary plot of the AE counts vs sustained load for all of the vessels subjected to sustained load tests. Note that the two vessels, K-42 and K-52, that did not show the induction periods (data on Figs. 9 and 13) display slopes that are significantly higher than the average slopes for the five vessels that did display induction periods prior to vessel failure.

The data suggest that AE monitoring of Kevlar 49/epoxy composite vessels can be developed into a technique to establish vessel integrity and to predict the residual service life of composite vessels subjected to a sustained load environment. Further work to study the combined effects of high prestress levels and pressurization dwell times on the performance of Kevlar 49/epoxy composite vessels is needed in order to provide assurance that composite vessels will meet long-term sustained load/cyclic fatigue requirements.

SUMMARY OF RESULTS AND CONCLUSIONS

The following major results and conclusions were obtained from the AE vessel data studied in this program:

1. The average of the AE signals sensed by the boss-mounted transducers on the vessels subjected to the sustained load tests were closely similar to each other. The AE data from the wall-mounted transducers on the same vessels varied widely and did not show any correlations with vessel performance.

2. The average AE signals from the boss-mounted transducers on the vessels subjected to sustained loading tests were independent of time-to-vessel failure. This observation indicates that a certain critical number of Kevlar 49 fibers must fracture prior to vessel failure.

3. Five out of the seven vessels subjected to sustained load tests displayed an increase in AE rate activity prior to vessel failure. This characteristic may be exploited to detect the incipient failure of composite vessels. These vessels displayed "induction" periods with high AE rates indicative of imminent failure.

4. The potential for using the AE transducer boss mounting technique evaluated in this program to monitor the integrity and service life of Kevlar 49 fiber/epoxy composite vessels is believed to be excellent.

REFERENCES

- [1] Lark, R. F., in Composites in Pressure Vessels and Piping, American Society of Mechanical Engineers, New York, 1977, pp. 17-49.
- [2] Hamstad, M. A. and Chiao, T. T., Society for the Advancement of Material and Process Engineering, Vol. 8, No. 1, Oct. 1976, pp. 31-45.
- [3] Hamstad, M. A., in Non-Metallic Materials Selection, Processing and Environmental Behavior, Society for the Advancement of Material and Process Engineering, Azusa, CA, 1972, pp. 321-331.
- [4] Hamstad, M. A. and Chiao, T. T., Journal of Composite Materials, Vol. 7, July 1973, pp. 320-332.
- [5] Hamstad, M. A. and Chiao, T. T., in Composite Reliability, Special Technical Publication-580, American Society for Testing and Materials, Philadelphia, PA, 1975, pp. 191-201.
- [6] Hoggatt, J. T., "Development of Cryogenic PRD-49-1 Filament-Wound Tanks," NASA CR-120835, National Aeronautics and Space Administration, Cleveland, OH, Dec. 1971.
- [7] Chiao, C. C., Sherry, R. J., and Chiao, T. T., Composites, Vol. 7, Apr. 1976, pp. 107-109.
- [8] Chiao, T. T. and Moore R. L., Journal of Composite Materials, Vol. 6, 1972, pp. 156-159.

TABLE 1. - FIBER AND RESIN MATERIALS

Fiber	Epoxy resin	Hardener	Gellation	Cure
Kevlar 49-III, ^a single-end, 285 filaments per end	DER-332 ^b 100 parts	T-403 ^c 39 parts	Room temperature (21° C) for 16-24 hours	3 hours at 90° C

^aE. I. Du Pont de Nemours & Co., Wilmington, Delaware.

^bDow Chemical Co., Midland, Michigan.

^cJefferson Chemical Co., Houston, Texas.

TABLE 2. - SINGLE-CYCLE BURST
STRENGTH TESTS

Vessel number	Burst pressure, GPa
K-32	0.01578
K-34	.01605
K-36	.01686
K-38	.01659
Average	0.01632 (2400 psi)

TABLE 3. - SUSTAINED LOADING/ACOUSTIC EMISSION TESTS OF 4-INCH
DIAMETER KEVLAR 49/EPOXY COMPOSITE VESSELS

[Note: Vessels pressurized to 0.01469 GPa (2160 psi) until failure occurred.]

Vessel number	Acoustic emission counts, total				Sustained load time to failure, h
	Location of AE transducer on vessel			Average total counts for top and bottom boss locations	
	Vessel side	Top boss	Bottom boss		
K-39	221×10 ³	36×10 ³	54×10 ³	45×10 ³	784
K-41	501	42	42	42	26
K-42	169	42	42	42	160
K-44	338	48	58	53	234
K-46	302	41	57	49	39
K-49	241	25	47	36	523
K-52	270	22	50	36	6

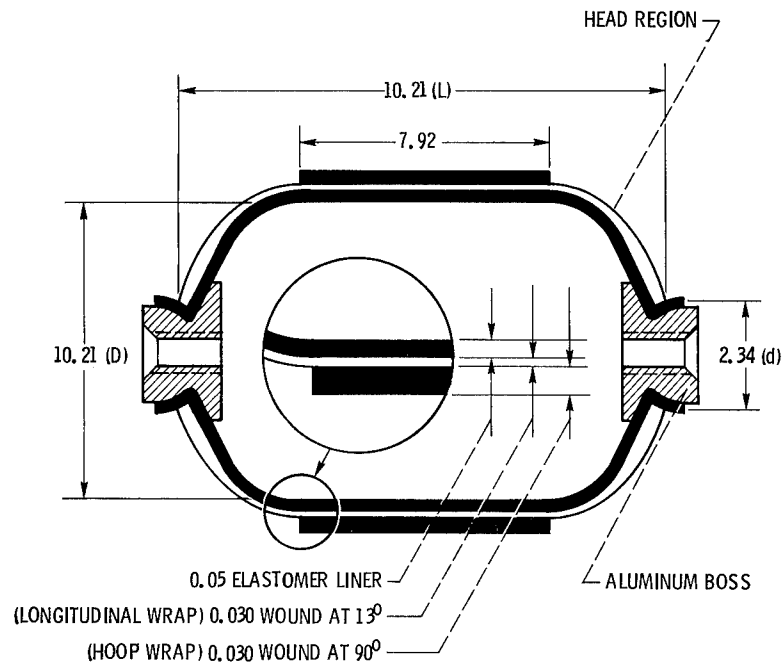


Figure 1. - Design of the 10.16 centimeters (4 in.) diameter Kevlar 49/epoxy composite pressure vessel. (Dimensions are in centimeters.)

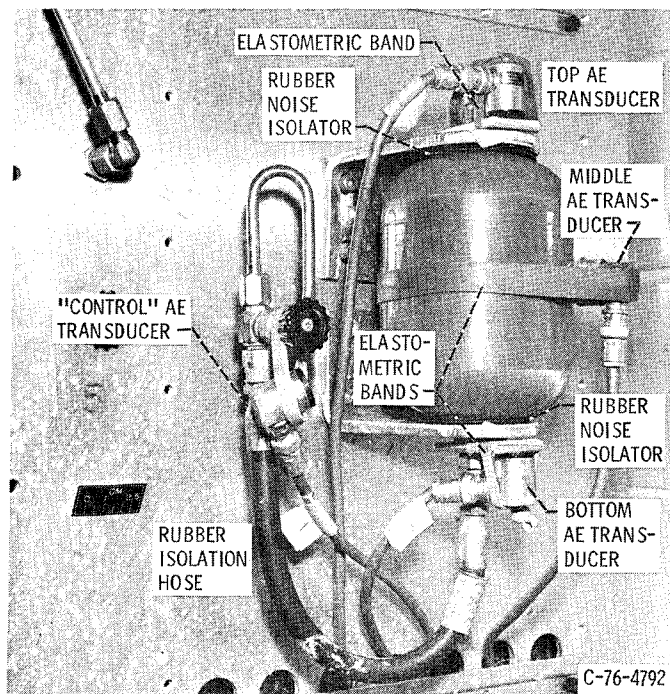
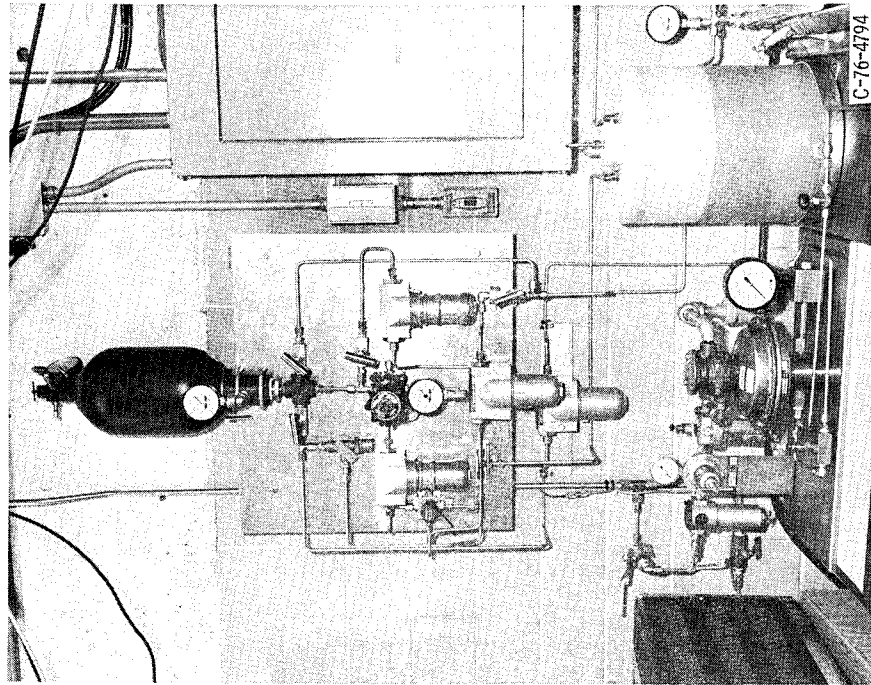
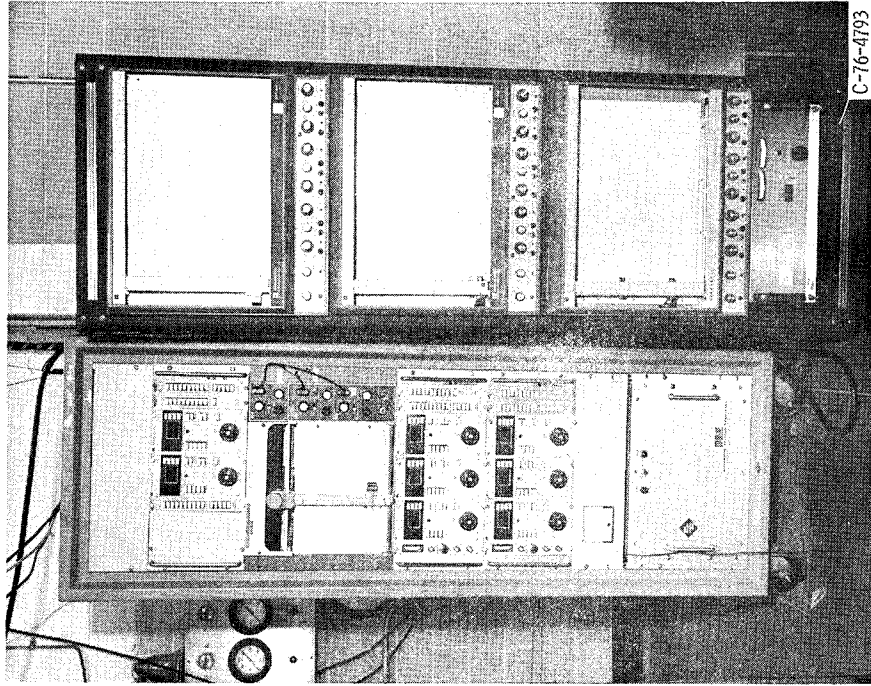


Figure 2. - View of Kevlar 49/epoxy composite vessel mounted in test fixture.



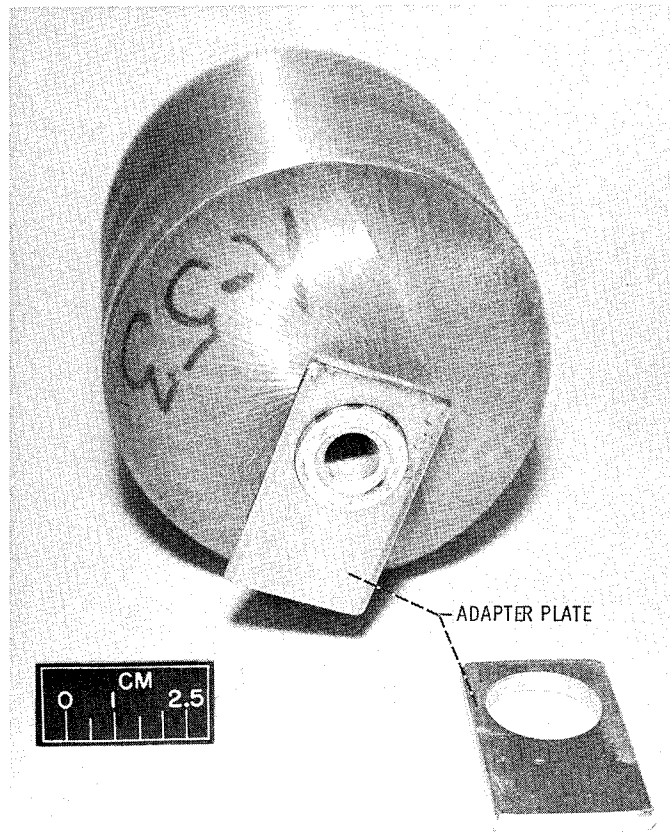
C-76-4794

Figure 3. - Hydraulic pressurization apparatus.



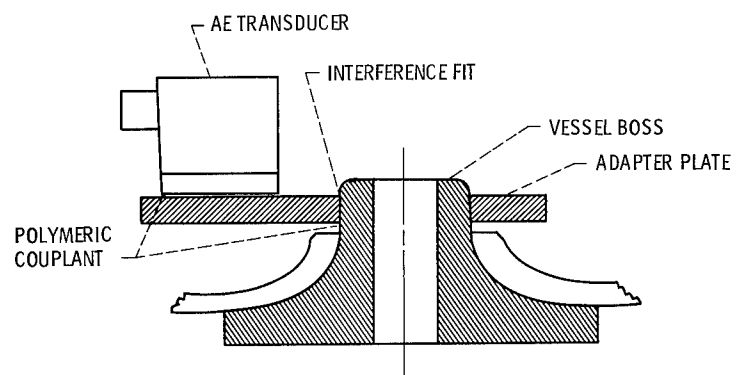
C-76-4793

Figure 4. - Acoustic emission data conditioning and recording apparatus.



C-76-4795

(a) END VIEW OF VESSEL AND BOSS WITH ADAPTER PLATE IN PLACE.



(b) CROSS SECTIONAL VIEW OF ADAPTER PLATE FASTENED TO VESSEL BOSS.

Figure 5. - Method for coupling AE transducers to vessel bosses.

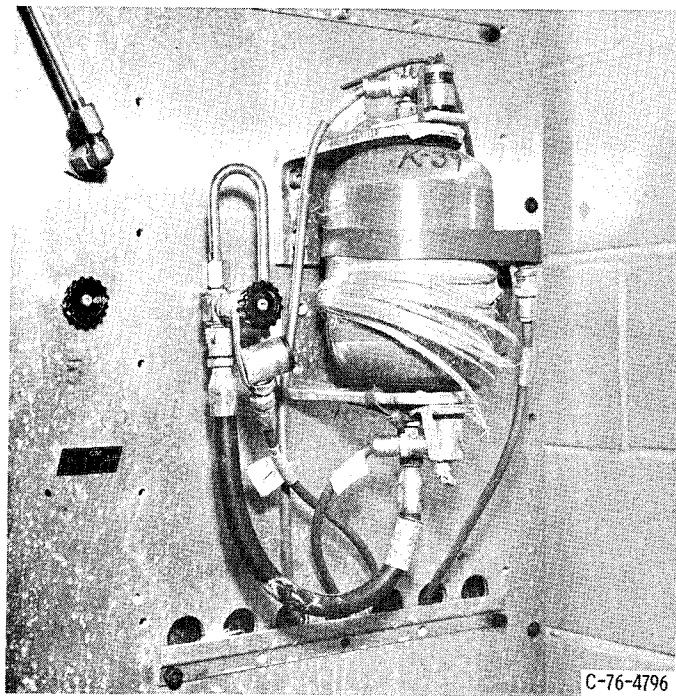


Figure 6. - View of typical vessel after failure.

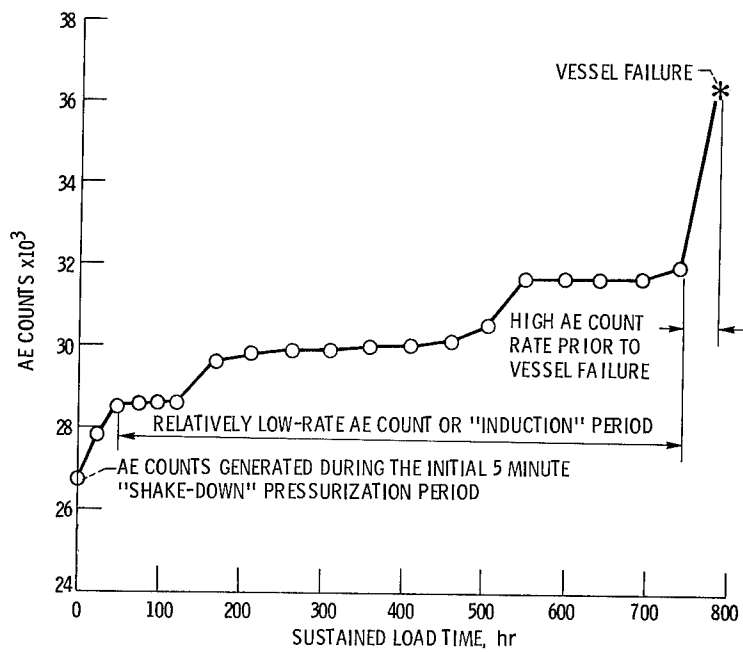


Figure 7. - Summation of AE counts vs sustained load time for vessel K-39.

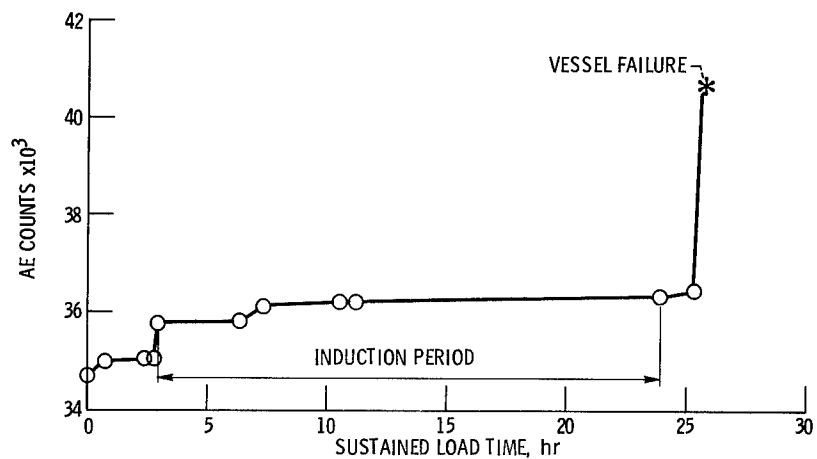


Figure 8. - Summation of AE counts vs sustained load time for vessel K-41.

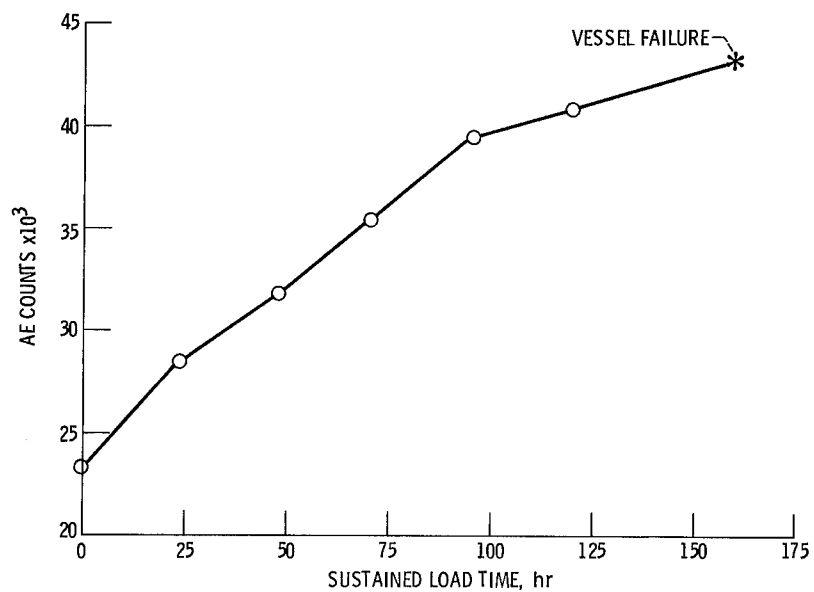


Figure 9. - Summation of AE counts vs sustained load time for vessel K-42.

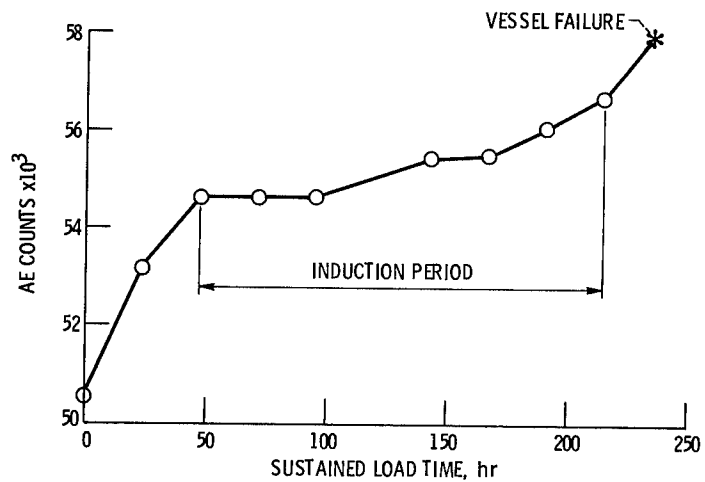


Figure 10. - Summation of AE counts vs sustained load time for vessel K-44.

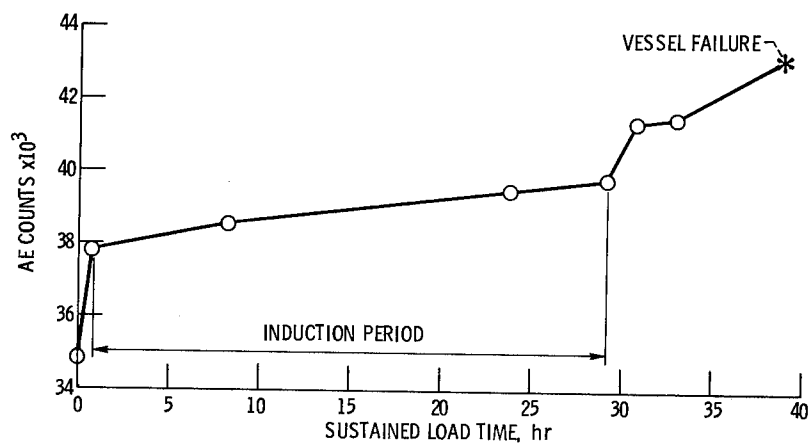


Figure 11. - Summation of AE counts vs sustained load time for vessel K-46.

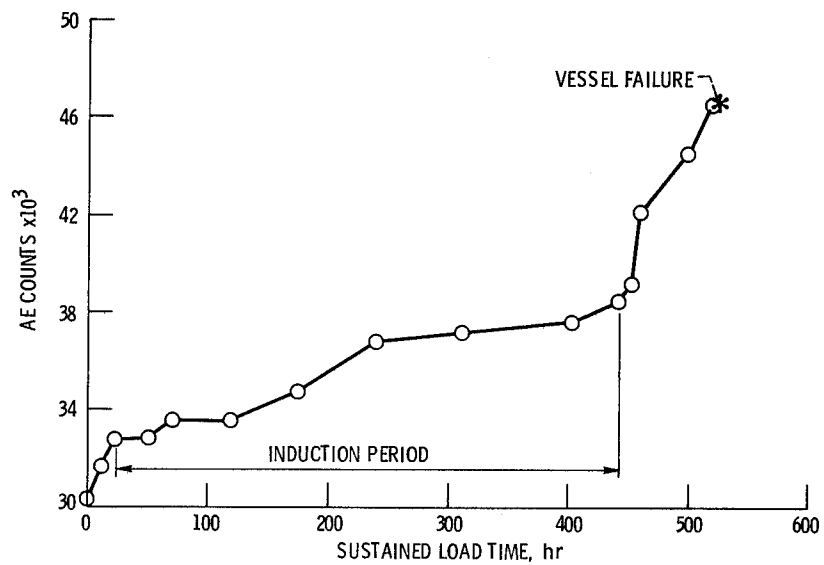


Figure 12. - Summation of AE counts vs sustained load time for vessel K-49.

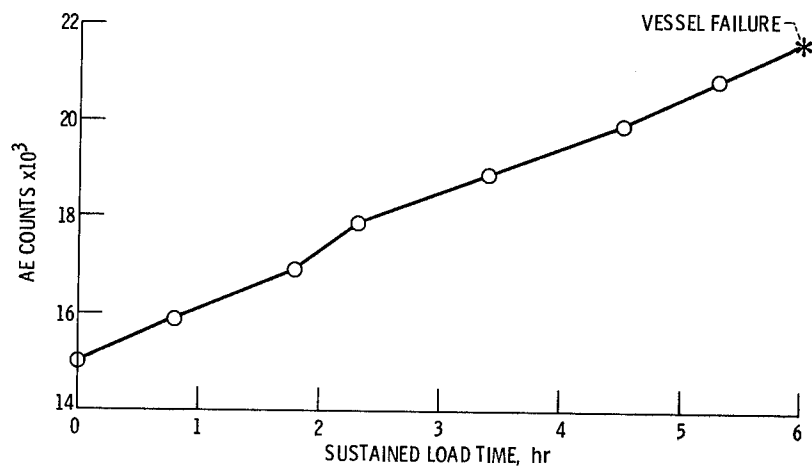


Figure 13. - Summation of AE counts vs sustained load time for vessel K-52.

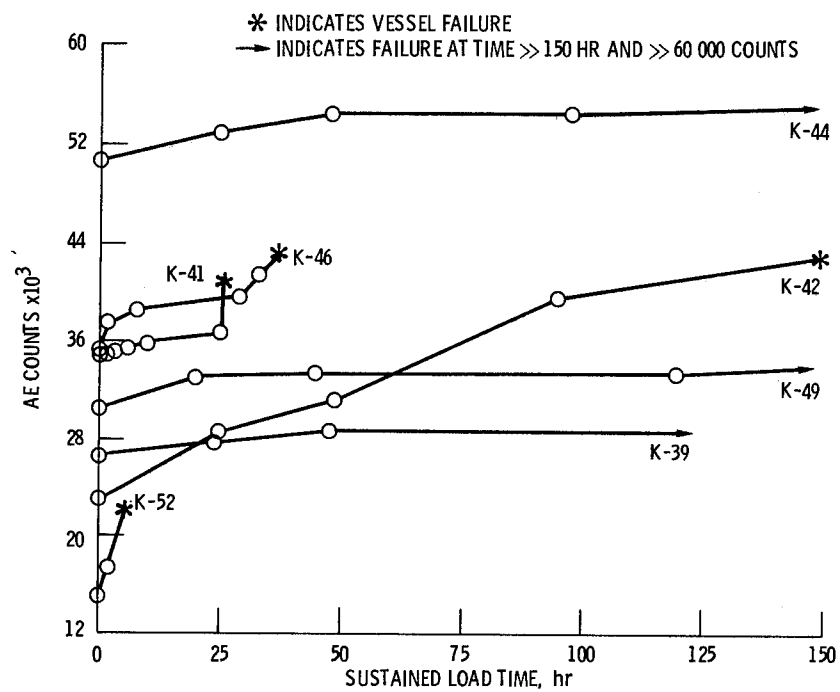


Figure 14. - Summation of AE counts vs sustained load time for all vessels.

1. Report No. NASA TM-78981		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle ACOUSTIC EMISSION TESTING OF COMPOSITE VESSELS UNDER SUSTAINED LOADING				5. Report Date	
				6. Performing Organization Code	
7. Author(s) R. F. Lark and P. E. Moorhead				8. Performing Organization Report No. E-9759	
				10. Work Unit No.	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135				11. Contract or Grant No.	
				13. Type of Report and Period Covered Technical Memorandum	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D. C. 20546				14. Sponsoring Agency Code	
15. Supplementary Notes					
16. Abstract <p>This paper describes a study of the acoustic emissions (AE) generated from Kevlar 49/epoxy composite pressure vessels subjected to sustained load-to-failure tests. The AE data from two different transducer locations on the vessels were compared. It was found that AE from vessel wall-mounted transducers showed a wide variance from those for identical vessels subjected to the same pressure loading. AE from boss-mounted transducers did, however, yield values that were relatively consistent. It appears that the signals from the boss-mounted transducers represent an integrated average of the emissions generated by fibers fracturing during the vessel tests. The AE from boss-mounted transducers were also independent of time for vessel failure. This suggests that a similar number of fiber fractures must occur prior to initiation of vessel failure. These studies indicate a potential for developing an AE test procedure for predicting the residual service life or integrity of composite vessels.</p>					
17. Key Words (Suggested by Author(s)) Acoustic emission; Acoustic emission induction period; Composite pressure vessels; Filament-winding; Kelvar 49/epoxy composites; Nondestructive tests; S-Glass/epoxy; Stress wave emissions; Sustained load to failure tests (creep rupture tests); Transducers			18. Distribution Statement Unclassified - unlimited STAR Category 24		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of Pages	
				22. Price*	